Dilution Refrigerator Operations

August 4, 2016

Contents

1 Overview

The dilution refrigerator is an integral component of the *HDice* target system. The goal of the system is the production and use of frozen-spin polarized deuterium hydride (HD) ice targets for nuclear physics experiments. The dilution refrigerator holds the HD ice target at the low temperature and high magnetic field required to produce polarization. In order to understand how the dilution refrigerator accomplishes its mission, a brief review of the target system is required.

1.1 HD Target

The current target design is for a 50 mm long by 15.0 mm diameter cylinder of HD ice. That cylinder is contained between two, 0.5 mm thick pCTFE cups, one inside the other. The outer one is 86.2 mm long and the inner one is 26.1 mm long. The inner, shorter cup is 26.2 mm OD and ends in a 45 degree cone with a 3 mm hole at its vertex. The 1.03 grams of HD liquid enters the target via this hole. The outer cup is enlarged to 29.7 mm ID in the region of the inner one to create a 1.75 mm gap. The gap is sealed at the top of the cups by the copper target support ring to which both cups are attached. Roughly 700 aluminum cooling wires, 51 microns in diameter are soldered to the support ring and extend through the gap down into the HD ice cylinder. The target support ring has a central clearance of 24.5 mm in order for the gamma ray beam to traverse the length of the target. The outer surface of the ring has a right-handed M35x1.0 thread for attachment of the ring into each of the four cryostats mentioned above. The inner surface of the ring has a left-handed M26x1.0 thread. This allows the transfer cryostat to attach to the ring with the left-handed inside thread and then unscrew the right-handed outside thread to release the target when the attachment screw of the transfer cryostat rotates counter-clockwise (as seen from above) or to attach the ring inside the appropriate cryostat and then detach from the transfer cryostat with clockwise rotation.

In order for the unsealed target not to sublime appreciably, its temperature must be maintained below 5 Kelvin, although brief excursions up to 7 Kelvin are tolerable. Table 1 gives the vapor pressure inside and sublimation rate from our HD target with its 3 mm hole.

Table 1: HD vapor pressure and sublimation rate vs. temperature

Temperature	Vapor Pressure	Sublimation Rate
7 K	7.8×10^{-4}torr	181 milli-gm/day
5 K	4.4×10^{-7}torr	123 micro-gm/day
4.216 K	3.9×10^{-9}torr	1.16 micro-gm/day

It is even more important to maintain the target at cold temperatures in order to reduce the rate of polarization loss as we shall see in the next section.

1.2 Target Cycle

The target is filled with HD by condensing HD vapor at just above the triple point of HD, 16.6 K. This temperature regime is reached with a specialized cryostat called the production cryostat or IceMaker, or by utilizing the vertical temperature gradient in the dilution refrigerator with its inner vacuum filled with exchange gas. After filling, the target is attached to a He3-He4 dilution refrigerator with a 10 mK base temperature and a 17 Tesla superconducting solenoid (DeepFreeze). With the aid of a 10^{-3} doping of metastable ortho-H2, the H reaches equilibrium polarization of up to 80% within two days. This polarization can be transferred to the D with an adiabatic fast passage technique and the H repolarized. Repeating the fast passage transfer allows D polarizations of up to 50% to be reached. After 6 weeks of aging the ortho-H2 has decayed to such an extent that the polarization approach to equilibrium is extremely slow. This allows the target to be removed from such extremes of temperature and magnetic field. The target is transferred to the storage cryostat (IceChest), where the target is stored and shipped between laboratories, and then to the inbeam cryostat (IceBucket), where the target is exposed to the gamma beam while surrounded by the detector array. After the target polarization has decayed too low to be useful, it is transferred to the production cryostat for vaporization of the HD and re-generation of the ortho-H2 at room temperature. The HD gas is then ready to begin a new cycle. Table 2 gives the polarization lifetime for a fully aged target as a function of temperature for both H and D at the nominal fields of the three relevant cryostats : storage, inbeam and transfer.

2 Dilution Refrigerator

Thus we see that dilution refrigerator must provide a 10 mK base temperature and a 17 Tesla magnetic field. Further, it must allow the target to be inserted and withdrawn. The refrigerator and magnet system is a semi-custom commercial device purchased from Oxford Instruments, a Kelvinox Model 1000. In the following subsections, each of the components of the dilution refrigerator will be examined in detail.

Table 2: Typical H and D polarization lifetimes vs temperature

Field	8 Tesla (storage)		0.8 Tesla (inbeam)		0.03 Tesla (transfer)	
Temperature	H	D	H	D	H	D
4.2 K	8 days	24 days			0.1 days	0.3 days
1.5 K	81 days	240 days	4 days	12 days	0.8 days	2.4 days
0.4 K			35 days	100 days		

See the two blueprint reproductions, Figures 1 and 2, and the two piping diagrams, Figures 3 and 4.

2.1 Outer Vacuum Chamber

The cryostat of the dilution refrigerator provides two insulating vacuum spaces. The one that insulates the liquid nitrogen and liquid helium reservoirs from room temperature is called the Outer Vacuum Chamber or OVC. There are no electrical feedthroughs into this volume. Pumpout is via a 25mm right angle valve, MS47, located on top of the cryostat. The valve incorporates a relief poppet designed to release at 4.4 psi. In addition, two large (68mm) relief flapper valves, S55 and S56, are located on the bottom of the cryostat.

2.2 LN2 Reservoir

Located inside the OVC is a torodial volume for liquid nitrogen, the LN2 reservoir. There are three access tubes symmetrically arranged on the top of the cryostat. One of the tubes is used for the level meter. It is sealed but carries a relief poppet, S51. The other two are used for filling and for venting the boiloff vapor. An aluminum radiation shield extends from the bottom of the LN2 reservoir and completely surrounds the LHe reservoir.

2.3 LHe Reservoir

A reservoir for liquid helium (LHe) forms the inside of the cryostat outer vacuum space. The 17 Tesla main magnet and its lambda point refrigerator are immersed in the reservoir. Two cold traps on the returning He3/He4 mixture are inserted into the reservoir. There are penetrations for the magnet leads, magnet electrical services, pumping of the lambda point refrigerator, control of the lambda needle valve, N63, and a helium level probe as well as the fill and two vent ports. The vents connects to the recovery section on the Auxiliary Panel of the Intelligent Gas Handling system. There is a quench relief on the vent, S54, which is an Oxford Instruments NW40 poppet set to release at 5.2 psi.

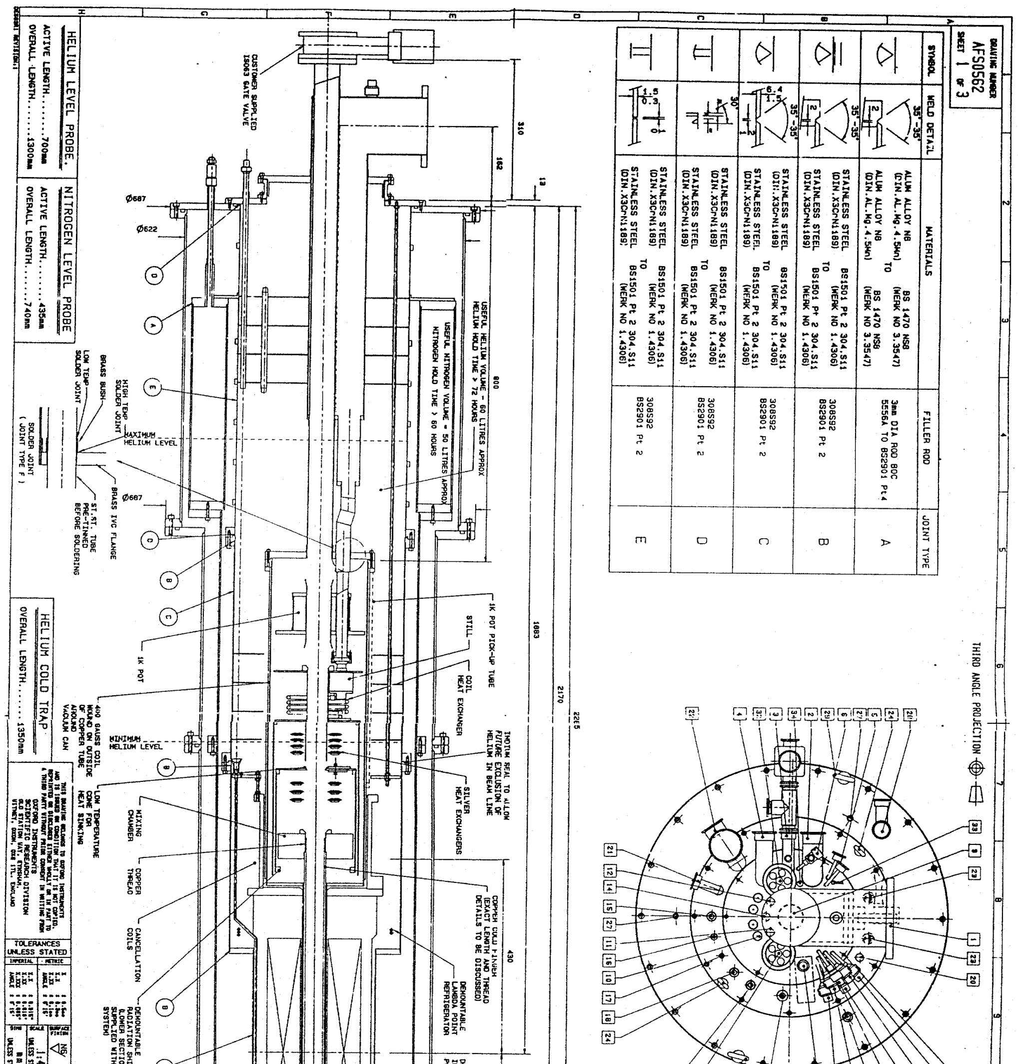
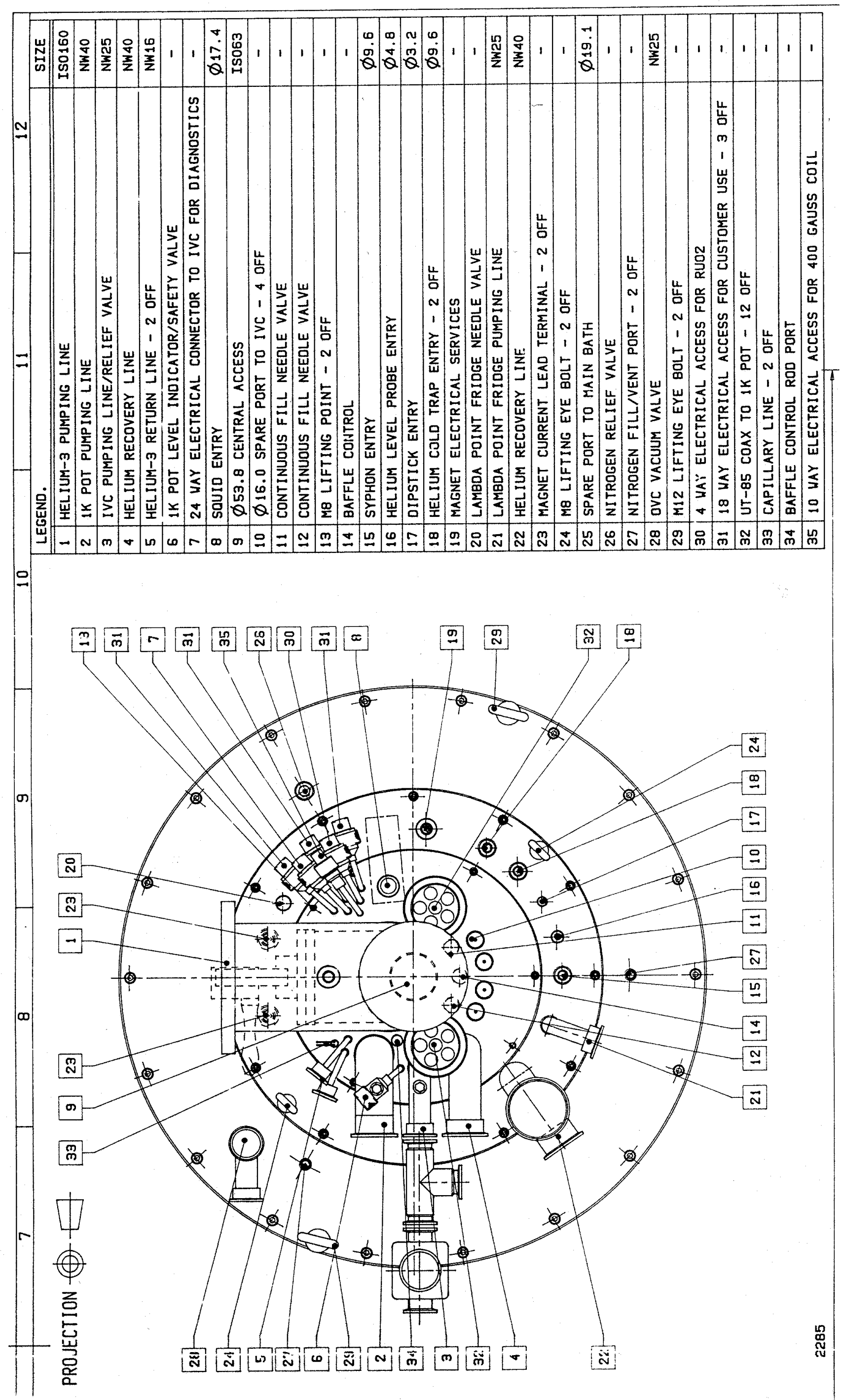


Figure 1: This a blueprint of the Dilution Refrigerator.



	LEGEND.	SIZE
1	HELIUM-3 PUMPING LINE	ISO160
2	1K POT PUMPING LINE	NW40
3	IVC PUMPING LINE/RELIEF VALVE	NW25
4	HELIUM RECOVERY LINE	NW40
5	HELIUM-3 RETURN LINE - 2 OFF	NW16
6	1K POT LEVEL INDICATOR/SAFETY VALVE	-
7	24 WAY ELECTRICAL CONNECTOR TO IVC FOR DIAGNOSTICS	-
8	SQUID ENTRY	Ø17.4
9	Ø53.8 CENTRAL ACCESS	ISO63
10	Ø16.0 SPARE PORT TO IVC - 4 OFF	-
11	CONTINUOUS FILL NEEDLE VALVE	-
12	CONTINUOUS FILL NEEDLE VALVE	-
13	M8 LIFTING POINT - 2 OFF	-
14	BAFFLE CONTROL	-
15	SYPHON ENTRY	Ø9.6
16	HELIUM LEVEL PROBE ENTRY	Ø4.8
17	DIPSTICK ENTRY	Ø3.2
18	HELIUM COLD TRAP ENTRY - 2 OFF	Ø9.6
19	MAGNET ELECTRICAL SERVICES	-
20	LAMBDA POINT FRIDGE NEEDLE VALVE	-
21	LAMBDA POINT FRIDGE PUMPING LINE	NW25
22	HELIUM RECOVERY LINE	NW40
23	MAGNET CURRENT LEAD TERMINAL - 2 OFF	-
24	M8 LIFTING EYE BOLT - 2 OFF	-
25	SPARE PORT TO MAIN BATH	Ø19.1
26	NITROGEN RELIEF VALVE	-
27	NITROGEN FILL/VENT PORT - 2 OFF	-
28	OVC VACUUM VALVE	NW25
29	M12 LIFTING EYE BOLT - 2 OFF	-
30	4 WAY ELECTRICAL ACCESS FOR RUO2	-
31	18 WAY ELECTRICAL ACCESS FOR CUSTOMER USE - 3 OFF	-
32	UT-85 COAX TO 1K POT - 12 OFF	-
33	CAPILLARY LINE - 2 OFF	-
34	BAFFLE CONTROL ROD PORT	-
35	10 WAY ELECTRICAL ACCESS FOR 400 GAUSS COIL	-

Figure 2: This a blueprint of the top of the Dilution Refrigerator.

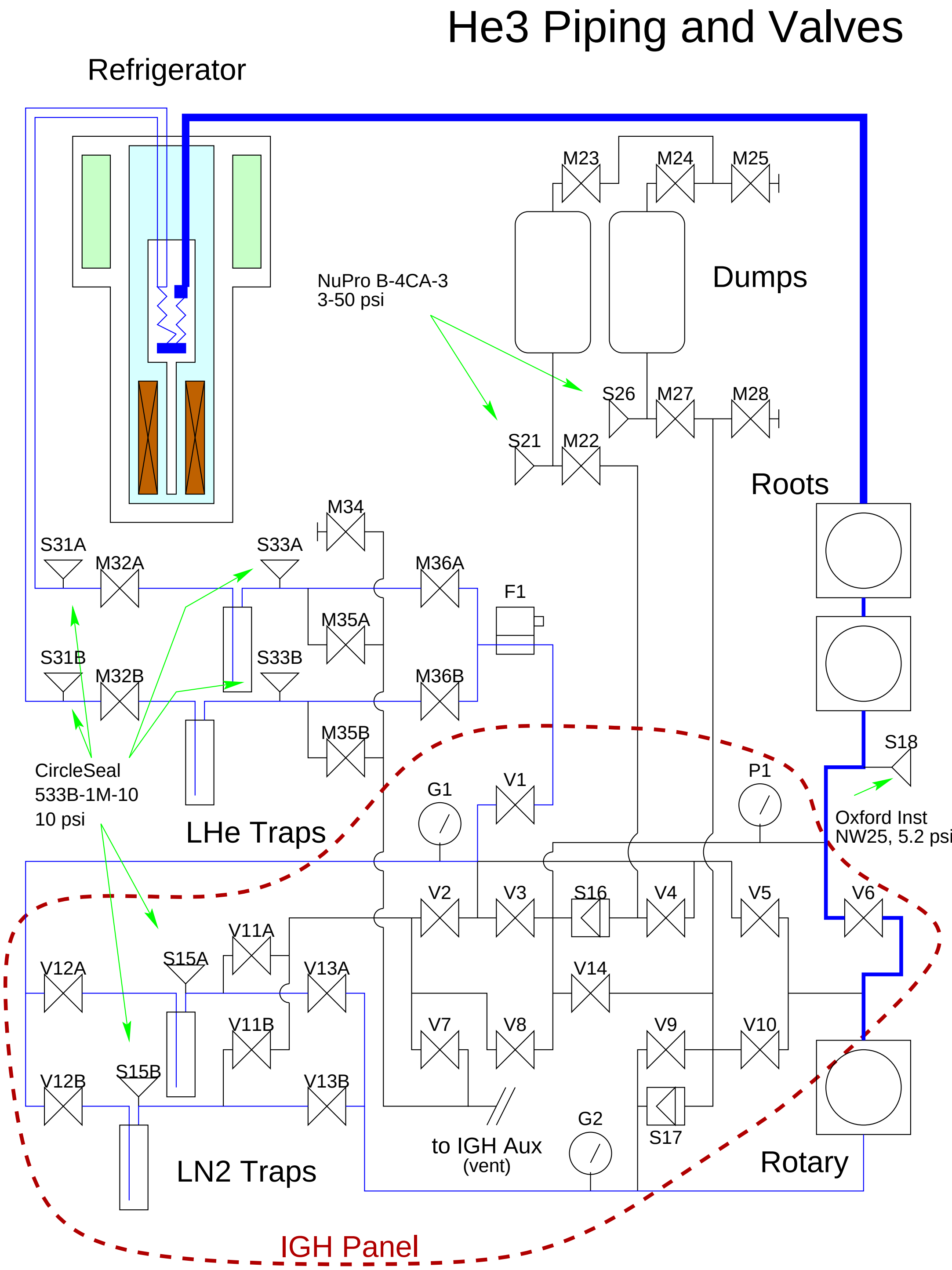


Figure 3: He3 piping and valves.

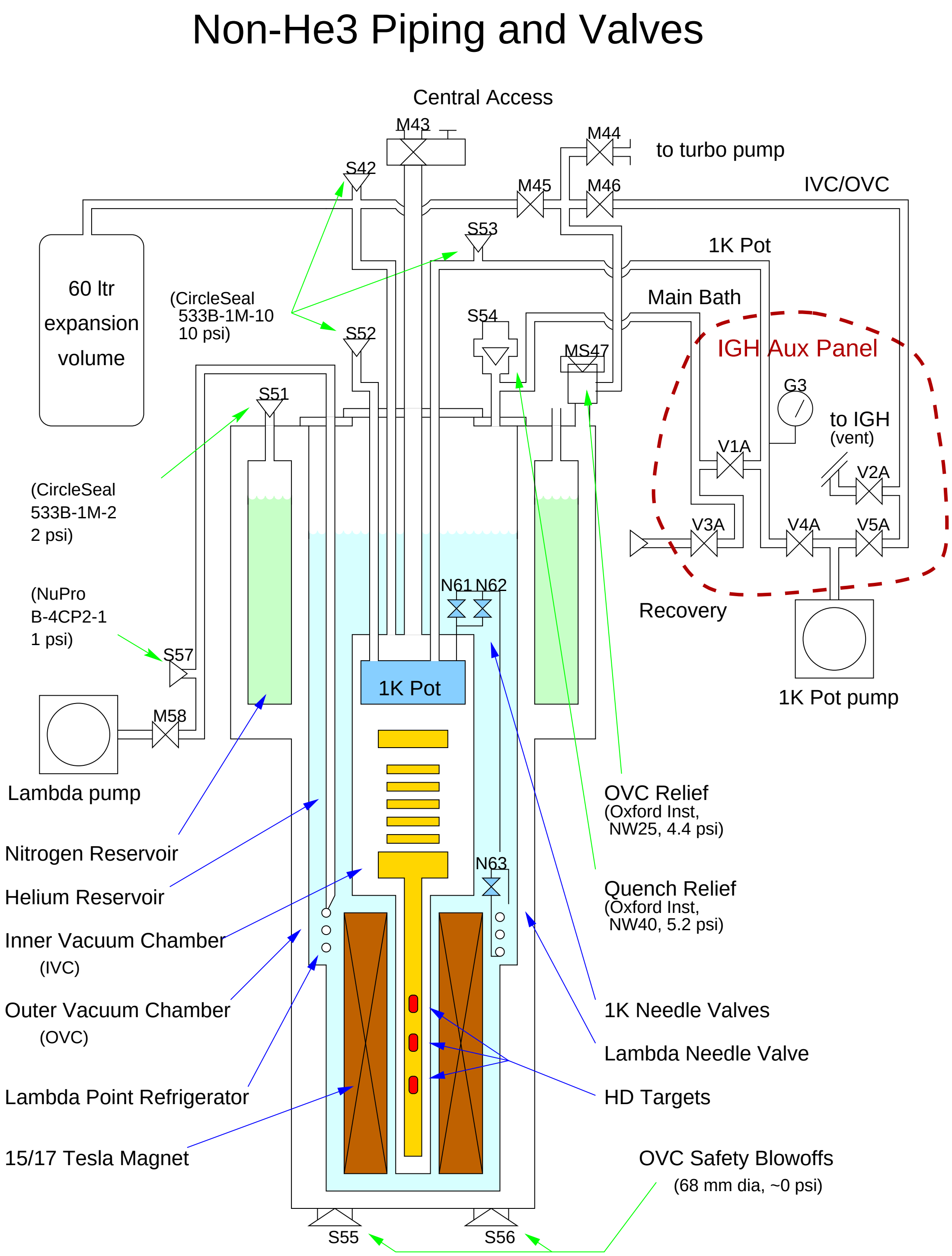


Figure 4: Non-He3 piping and valves.

2.4 Superconducting Main Magnet

A superconducting magnet is mounted in the bottom of the LHe reservoir. The magnet is a combination NbTi and NbSn solenoid with a 55 mm bore. It has a maximum central field of 15 Tesla with 94.505 amps at 4.2 K and 17 Tesla with 107.106 amps at 2.2 K. The magnet has an inductance of 111 Henries and hence a stored energy at full field of 0.50 MJ (191 liters of LHe) at 4.2 K and 0.64 MJ (245 liters of LHe) at 2.2 K. This stored energy is more than enough to boil the entire LHe reservoir. In addition the eddy current heating of the dilution unit will result in the boiling out of most of the mixture. The power supply for the magnet is located in the system control cabinet.

The 2.2 K temperature is produced by a lambda point refrigerator located on the top of the magnet. It consists of a spiral coil of tubing fed by a needle valve, N63, and pumped by a 65 m3/hr rotary pump. A control unit for the valve and the pump is located in the system control cabinet.

2.5 Superconducting Transfer Solenoid

A superconducting coil has been wound on a copper sheath on the outside of the Inner Vacuum Chamber (IVC). This provides a small field to augment the fringe field of the main solenoid for holding the target polarization during the transfer between the main solenoid and the high Tc magnet located in the LN2 reservoir of the transfer cryostat (stopped at the shutter opener on the 1K pot).

2.6 Inner Vacuum Chamber

The other insulating vacuum space of the dilution refrigerator is a volume suspended in the LHe reservoir called the Inner Vacuum Chamber or IVC. It insulates the 1K pot and the dilution unit itself from the 4.2 Kelvin liquid helium. Besides a number of tubes carrying electrical wiring there are two main penetrations into this volume. One is the central access tube which is closed at the top by the ISO LF63 gate valve M43. The other is the standard pumpout port which branches to NW25 valve M45, to 10 psi relief poppet S42 and to a 60 liter expansion volume which captures the HD target material in the event that cooling is lost and the HD vaporizes.

2.7 1K Pot

The 1K Pot is a copper canister within the IVC that is feed with LHe from the LHe reservoir via either of two needle valves, N61 or N62, and is pumped by a 65 m3/hr rotary pump through valve V4A located on the IGH Auxiliary panel. It produces a temperature of roughly 1.2 Kelvin that is used to condense the returning concentrated He3/He4 mixture. A separate tube carries electrical connections for a level meter in the pot and is protected by 10 psi relief poppet S52. The pump line is protected by 10 psi relief poppet S53.

2.8 Dilution Unit

The heart of the dilution refrigerator is its dilution unit. It is mounted below the 1K Pot and is part of the circulating He3/He4 system. It consists of the mixing chamber, seven step heat exchangers, two continuous heat exchangers and the still. Mixture is pumped from the unit through the ISO LF160 pumping line by the 1200 m3/hr roots pumping system and returned through either of two condenser lines as determined by valves M32A and M32B. A copper tail extension from the mixing chamber holds 3 HD targets in the bore of the main solenoid.

2.9 Ancillary Equipment

2.9.1 Pump Stand

A 1200 m3/hr Roots blower, a backing 250 m3/hr Roots blower, and a 65 m3/hr sealed rotary vane pump form a pumping system for the circulating helium3/helium4 mixture. They are mounted on a separate stand. In addition, a 65 m3/hr rotary pump for the 1K pot and a 65 m3/hr rotary for the lambda point refrigerator are also located on the stand. Next to the stand are the two 230 liter tanks used for storing the 354 liters of He3/He4 gas mixture. Along side is the power distribution panel for the pumps with resets for overloads and over pressure.

2.9.2 Control Cabinet

Most of the sensor readouts, valves and controls are contained in a central control cabinet that houses the Intelligent Gas Handling (IGH) system, the IGH Auxillary panel, the Intelligent Power Supply (IPS) and the lambda fridge controller for the magnet. The rack is coupled by GPIB to a monitoring and control computer running LabView.

2.9.3 Expansion Volume

A 60 liter tank has been added to provide a total expansion volume sufficient for 3 HD targets in the event that cooling is lost and the HD vaporizes. This volume plus the volume of the inner vacuum chamber (IVC) itself is sufficient to maintain the pressure below atmospheric. It is coupled to the IVC by a 2.4 meter long, 1.9 cm diameter, flexible hose and is located in the pit.

3 Procedures

Before attempting any of the procedures detailed in the following sections, all personnel should acquaint themselves with standard laboratory safety procedures, including vacuum pumping, liquid cryogen handling, magnetic field safety and proper hoist operation. All personnel must have read the manuals provided by Oxford Instruments and this manual fully, and must be certified by the head of the SPHICE target group at BNL. This equipment is for experimental use only and requires handling by trained personnel only.

3.1 Preparing for Cooldown

This procedure details preparing the refrigerator for operation.

1. Attach a turbo pumping station to the KF40 flange of diaphram valve M44 on the top of the cryostat. Close M44. start the roughing pump, open the station valve and put the turbo in standby. Close valves M45, M46 and MS47. Open valve M44 to rough out the manifold. Then open MS47, take the turbo pump out of standby, and evacuate the cryostat outer vacuum chamber space (OVC) to better than 5×10^{-6} torr. Because of the large amount of super insulation in the OVC, pump for at least 24 hours. Close MS47.

2. Close M44 and put the turbo in standby. Close valves M45, M46 and MS47. Open valves M44 and M45 to rough out the cryostat inner vacuum chamber space (IVC). Then take the turbo pump out of standby, and evacuate the IVC to better than 5×10^{-6} torr. Close M45 and M44. Shutdown and detach the pumping station.

3. Attach a leak detector to M44. When the unit is ready, open M44 and MS47. Confirm the helium level in the OVC is below 10^{-8} mbar l/s. Close MS47.

4. Open M45. Confirm the helium level in the IVC is below 10^{-8} mbar l/s. Close M44 and M45.

5. Attach a supply of standard purity He4 gas to the IGH vent valve, M34. Close V7, M35A, M35B, M46, V1A, V2A, V3A, V4A, V5A, N61 and N62. Start the 1K pot pump.

6. Open valves V5A and V2A. Alternately open the He4 gas supply and M34 to purge the connecting line. Close M34 and leave the supply open.

7. *Check for leaks from the 1K pot.* Open valve V4A and pump out the 1K pot to better than 1 mbar. Connect the IVC to the leak detector by opening valves M44 and M45. Turn off the the 1K pot pump and back fill the pot to 1 bar from the helium gas supply via M34 while monitoring the leak detector signal for leaks into the IVC. Close M34.

8. *Check for leaks from the helium reservoir.* Re-start the 1K pot pump and open valve V3A to pump out the 1K pot and the helium reservoir to better than 1 mbar. Turn off the 1K pot pump and back fill the the pot and the reservoir to 1 bar from the helium gas supply via M34 while monitoring the leak detector for leaks into the IVC and the OVC by alternately opening and closing valves M45 and MS47. Close V1A and M34. Open V3A. Detach the standard purity helium gas supply.

9. *Check the operation of the 1K pot needle valves.* Re-start the 1K pot pump and pump out the 1K pot. Close V4A. Open the remote control needle valve N61 and note the pressure rise in the pot. Close N61. Open the manual needle valve N62 and note the pressure rise in the pot. Close N62 and open V4A to pump out the pot again. Close V4A. Shutoff the 1K pot pump.

10. *Check the operation of the Lambda fridge needle valve.* Close valves M58 and remote control needle valve N63. Start the Lambda pump. Open M58 and evacuate the Lambda fridge to better than 1 mbar. Close M58. Open N63 and note the pressure rise. Allow the pressure to rise to 1 bar and then close N63. Repeat. Turn off the Lambda pump.

11. *Check the dilution unit for leaks and throughput.* Attach a supply of high purity He4 gas to the IGH vent valve, M34. Close V7, M35A, M35B, M46, V2A, V4A, V5A. Start the 1K pot pump.

12. Open valves V2A and V5A. Alternately open the He4 gas supply and V34 to purge the connecting line. Close M34, V2A, and V5A and leave the supply open.

13. Close V1, V4, V5, V6, V8, V9, V14, M32A, M32B, M35A, and M35B. Open M36A and M36B. Open V7, V2, and V3, then use M34 to fill the still to 1 bar, monitoring the pressure on G1. Close V2, V3, V7 and M34. Close the high purity helium supply.

14. Open V1 and M32A. Note the steady pressure rise on G1, which should be 17 mbar/minute. Close M32A and open M32B. Again note the pressure rise, which should also be 17 mbar/minute. Open V3, M32A, and M32B to equalize the pressures.

15. Connect the IVC to the leak detector by opening valves M44 and M45. Check the level of helium in the IVC on the leak detector. Close M44 and M45. Shutdown the leak detector and remove it.

16. Open V1, V2, V3, V2A, V5A, M32A, M32B, M36A, and M36B to pump out the unit with the 1K pot pump. When the pressure is below 1 mbar, close V1, V2, V3, V2A, V5A, M32A, M32B, M36A, and M36B. Shutoff the 1K pot pump.

17. *Add exchange gas to the IVC.* Open V2A, M45 and M46. At the supply end, briefly open to air the 5/16 inch flexible tubing on the high purity gas line to reduce its pressure to 1 bar. Pinch off the tubing 10 cm from M34 and then open M34 briefly to add 5 cc's of helium to the IVC. Close V2A, M45 and M46.

At this point, the system is ready to be cooled to LN2 temperature.

3.2 Cooling to LN2

This procedure details cooling the cryostat from room temperature to that of liquid nitrogen, 77 Kelvin. It assumes that the Procedure 3.1 has been preformed prior to beginning this procedure. Note that this procedure requires six to eight hours to complete.

1. Insert the LN2 blow-out tube into the syphon entry port and connect a LN2 supply dewar to it. Detach the line going to helium recovery or the check valve from valve V3A. Slowly open the valve on the LN2 supply dewar and adjust it to produce a steady light breeze out the recovery port. Record the time and readings in the logbook.

2. Monitor the progress of the magnet cooling by measuring its resistance with an ohm meter across the leads of the magnet. The magnet should be 49.5Ω at room temperature and drop to 40.9Ω at 77 K. The magnet should be cooled no faster than a tenth of an Ohm every 5 minutes during the initial phase. Adjust the flow rate on the LN2 as necessary.

3. Roughly every hour check the operation of the 1K needle valves by performing Procedure 3.1, step 9 and check the operation of the lambda fridge needle valve by performing Procedure 3.1,step 10.

4. After roughly six to eight hours the magnet should have dropped to 40.9Ω, indicating it is at LN2 temperature and liquid should have begun accumulating. One may now safely increase the rate of transfer. Accumulate liquid to at least 10 cm above the magnet (or about 135 cm below the dipstick entry). This can be checked by briefly inserting a 3 mm diameter plexiglass rod into the dipstick entry port, pulling it out and noting the point on the rod where the subsequent condensation occurs. Record the time and readings in the logbook.

5. Wait an additional hour of cooling, before starting the next step, transferring the LN2 from the LHe reservoir to the LN2 reservoir.

6. Connect between the LN2 blow-out tube and one of the LN2 ports with neoprene tubing. Use temporary tubing to direct the exhaust from the other LN2 port away from and down below the top plate (the third port has the level meter). Connect a helium gas supply cylinder to the recovery port on valve V3A. Open the shutoff valve on the helium supply cylinder and adjust the regulator to pressurize the LHe reservoir with 2 to 3 psig of helium. This will force LN2 out of the LHe reservoir and into the LN2 one. Record the time and readings in the logbook.

7. Repeat the throughput checks on the dilution unit as given in steps 11, 12, 13, 14, and 16 of Procedure 3.1. The throughputs should now be 120 mbar per minute.

8. When all the liquid has been transferred, there will be a sudden drop in helium pressure on the delivery side of the regulator on the helium gas supply and an increase in flow. Continue flowing gas for another 3 to 5 minutes to remove the last drops of liquid. Close V3A. Record the time and LN2 level readings in the logbook. Remove the connection between the LHe and LN2 reservoirs. Stopper the LN2 fill port (allowing it to continue to exhaust through the other port).

9. Remove the LN2 blow-out tube and seal the syphon entry port. Close V1A, V3A, V4A, and V5A. Turn on the 1K pot pump. Open V1A and V4A, and evacuate the LHe reservoir to better than 0.1 mbar. If the pumping pauses, it probably means there is still some LN2 in the LHe reservoir. This must be removed by repeated flush and pump cycles. Note, while doing this step, also perform Procedure 3.5 to top off the LN2 reservoir.

10. Close V4A and open V1A and V3A to back fill the pot and the LHe reservoir with helium gas.

11. Close the shutoff valve on the helium gas supply and remove the helium gas supply. Re-attach the line to the helium recovery system or the check valve to V3A.

The cryostat has now been pre-cooled to LN2 temperature and is ready to be cooled to LHe temperature.

3.3 Cooling to LHe

This procedure details cooling the cryostat from 77 Kelvin to that of liquid helium, 4.2 Kelvin. This procedure requires that Procedure 3.2 has been completed immediately prior to beginning this procedure.

1. If necessary, perform Procedure 3.5 to top off the LN2 reservoir.

2. If the check valve is on V3A, detach it.

3. Attach the helium supply lance end to the liquid helium supply dewar entry and allow the dewar boiloff to purge the transfer tube and engage the cone that directs the flow below the magnet. Un-stopper the syphon entry port and insert the helium fill lance end of the transfer tube. Slowly insert the helium supply lance end into the liquid helium supply dewar and begin transferring at a rate of 1 to 2 liters of gas per minute. This should generate a barely perceptible breeze out the exhaust tubing.

4. Monitor the progress of the cooling by measuring the resistance of the magnet. with an ohm meter across the leads.

5. After roughly 2 hours the magnet should go superconducting, zero resistance, and liquid should begin accumulating. Increase the rate of transfer and fill the reservor to 100% as measured on the level meter. Be sure to set the meter for high rate readings while filling.

6. Power the sorb heater to 10 mW or higher to raise the sorb temperature to 30 K or higher. The controls for the power supply are on the Intelligent Gas Handler panel.

7. Remove the fill lance and plug the port. Set the level meter for low rate readings. Record the time and cryogen levels in the log book.

8. After an additional hour, the blowoff will have stablized. Re-attach the check valve to V3A if it is in use.

The cryostat is now at LHe temperature and can remain so indefinitely as long as the cryogens are replenished. The system is ready to proceed to transfer in HD ice targets made in the Production dewar using Procedure 3.19, or to make HD ice targets using Procedure 3.22, or to cool down to base temperature using Procedure 3.4

3.4 Run to Base

This procedure describes the automated cooling of the cryostat from LHe temperature to the base temperature of the dilution refrigerator. This procedure requires that Procedure 3.3 has been completed prior to beginning this procedure.

1. Start the LabView control software for the refrigerator on the control computer. This can be done by clicking on the Oxford Instr icon on the desktop. Select the Applications menu in the oimenu.vi window and click on the Kelvinox button. Note that the valves, temperatures, etc can be monitored and controlled by clicking on the fridge front panel button of the KelvFrontPanel.vi window, that appears.

2. If and only if this is the first run-to-base since cooldown, so that all the mixture is in the dumps, click on the 4.2K Cooldown button of the KelvFrontPanel.vi window. The computer will warm the sorb pump on the 1K pot to release the helium exchange gas (if any is present) and pump the 1K pot and circulation lines. It then waits for the the mixing chamber temperature to reach 5K when it then turns the sorb heater off and puts some liquid in the pot to maintain the pot below 6K and the sorb below 10K. After waiting 20 minutes for the sorb to pump the helium exchange gas, the sequence exits.

3. Perform Procedure 3.5 to top off the LN2 reservoir and to fill the LN2 trap dewar. Insert the LN2 traps into their dewar. Insert the LHe traps into the LHe reservoir.

4. Open the dump valves M22 and M27.

5. Click on the Run-to-Base button of the KelvFrontPanel.vi. The computer will fill and pump the 1K pot, condense in the mixture, start circulating the mixture and then exit the sequence.

6. Note the time, trap in use, cryogen levels, etc. in the log book.

The dilution refrigerator will continue to cool to base and remain there provided that the liquid cryogens are replenished with Procedures 3.5 and 3.6 as needed, that the LN2 traps are switched and cleaned with Procedure 3.7 whenever the G1 to G2 pressure difference becomes too large and that the LHe traps are switched and cleaned whenever the pressure on G1 beccomes too high.

3.5 Replenishing LN2

This procedure details refilling the liquid nitrogen reservoir.

1. Remove the plug from the LN2 reservoir inlet and insert the transfer hose.
2. Open the valve on the LN2 supply cylinder and begin the transfer.
3. When the LN2 level meter indicates 100% stop the transfer, remove the transfer hose from the reservoir inlet and replace the stopper.
4. If the LN2 traps are in use, lift the top cover of their dewar and insert the transfer hose. Fill the dewar.
5. Note the date, time, starting and ending level readings in the log book.

3.6 Replenishing LHe

This procedure details refilling the liquid helium reservoir. This procedure requires that the cryostat be in normal operating condition following Procedure 3.3.

1. If using a small check valve on the recovery port of valve V3A, remove it.
2. Begin inserting the withdrawl lance end of the transfer tube into the LHe supply dewar When the blowoff begins to increase, stop inserting the lance and allow the tube to be cooled by the blowoff until the characteristic white flame appears at its tip, signaling that it is at liquid helium temperature. Relieve the pressure by opening the valve on the helium supply dewar.
3. WARNING: DO NOT insert the tube below the liquid level or engage the cone that directs the flow below the magnet. Insert the fill lance end of the transfer tube into the syphon entry port of the cryostat for a distance of 51 cm or 20 inches. Begin transferring by re-pressuring the supply dewar.
4. Monitor the progress of the fill with the LHe level meter. Note that the meter should be switched to high rate readings in order to have prompt response.
5. When the meter indicates full, relieve the pressure on the supply dewar, remove the fill end of the tube from the cryostat and seal the syphon entry port. Remove the withdrawl end of the tube from the supply dewar. Return the level meter to low rate mode.

6. When the boiloff has settled down, replace the small check valve on the recovery port of valve V3A if it was removed in step 1.

7. Note the date, time, starting and ending level readings in the log book.

The LHe has now been replenished.

3.7 Cleaning an LN2 Trap

This procedure details cleaning one of the two liquid nitrogen cooled traps in the small dewar next to the pumping cabinet. This procedure assumes that the cryostat is in normal operating condition following Procedure 3.4. This procedure is indicated when the pressure drop through the trap, that is the difference between gauges G1 and G2, becomes too large.

1. Make sure the alternate clean trap has its cleanout valve(V11A or V11B) closed and that the trap is inserted in the LN2 reservoir.

2. Switch the circulation from the blocked trap to the alternate by clicking the switch trap button on the KelvFrontPanel.vi. Note the trap change in the log book.

3. Connect a roughing pump to manual valve M34 and pump out the manifold between valves M34, M35A, M35B, and V7 by opening M34.

4. *Scavenge the mixture from the dirty trap.* Close M34 and V7 and then open V8 and the blocked trap pumpout valve (either V11A or V11B) to pump the mixture in the trap back into circulation.

5. After several minutes of pumping, close V8 and open to the roughing pump with V7 and M34. Now lift the trap from the LN2 reservoir. Block the hole in the LN2 cover to prevent icing.

6. Allow the trap to slowly warm while being pumped by the roughing pump. In about 20 minutes, the trap will release its load of air and other contaminants and the pump will gurgle. Continue to pump for at least an hour after the trap has reached room temperature. This process can be speeded by warming the trap with a hot air gun but do not exceed 100 C on the trap as this may damage the charcoal.

7. Close the cleanout valve on the trap (either V11A or V11B) and close valves V7 and M34. Disconnect the roughing pump. Place the trap back in the LN2 container.

At this point the trap is ready to return to service whenever desired.

3.8 Cleaning an LHe Trap

This procedure details cleaning one of the two liquid helium cooled traps in the helium reservoir. This procedure assumes that the cryostat is in normal operating condition following Procedure 3.4. This procedure is indicated when the input pressure to the refrigerator, that is gauge G1, becomes too large.

1. Make sure the alternate trap has its manual cleanout valve(M35A or M35B) closed and that the trap is inserted in the LHe reservoir.

2. Switch the circulation from the blocked trap to the alternate by opening the in and out valves on the clean trap (M32A and M36A, or M32B and M36B) and then closing the corresponding valves on the dirty trap. Note the trap change in the log book.

3. Connect a roughing pump to manual valve M34 and pump out the manifold between valves M34, M35A, M35B, and V7 by opening M34.

4. *Scavenge the mixture from the dirty trap.* Close M34 and then open V7, V8. Slowly open the blocked trap pumpout valve (either M35A or M35B) to pump the mixture in the trap back into circulation without disturbing the pressure.

5. After several minutes of pumping, close V7 and V8 and open to the roughing pump with M34. Now lift the trap from the LHe reservoir. Block the port on the LHe reservoir to prevent icing.

6. Allow the trap to slowly warm while being pumped by the roughing pump. In about 2 minutes, the trap will release its load of contaminants and the pump will gurgle. Continue to pump for at least 10 minutes after the trap has reached room temperature. This process can be speeded by warming the trap with a hot air gun .

7. Close the cleanout valve on the trap (either M35A or M35B) and close valve M34. Disconnect the roughing pump. Place the trap back in the LHe reservoir.

3.9 Idling the Cryostat

This procedure stops the circulation, recovers the mixture and warms the 1K pot to 4.2 K. It assumes the system is circulating mixture after Procedure 3.4.

1. *Recover the mixture.* Open V9. Close V13A and V13B.

2. *Stop the 1K pot.* Close needle valves N61 and N62 and close V4A. Open valve V1A to vent the pot to the LHe reservoir. Close V5A and stop the 1K pot pump.

3. Monitor the pressure on G2 and use V6 to trottle the rate of recovery.

4. While monitoring the temperatures of the still and mixing chamber, raise their heaters to maximum to speed the recovery of the mixture. Turn off the appropriate heater when the temperature at that point rises above 2 Kelvin.

5. When the pressure reading on G2 matches the initial value and the pressure on P1 is less than 0.1 mbar, close valves V4, V6, V9, V12A and V12B. Open V3. Stop the Roots pumps and the Rotary pump.

3.10 Warming up the Cryostat

This procedure stops the circulation, recovers the mixture and warms the cryostat to room temperature. It is required that both magnets be de-energized (see Procedures 3.16 and 3.18).

1. Recover the mixture using Procedure 3.9 or click on the Warm button of the KelvFrontPanel.vi window.

2. When all the mixture has been recovered, the traps are not needed and the cleanout Procedures 3.7 and 3.8 should be performed on them,

3. Stop replenishing the cryogens and allow the system to warm. Depending on the cryogen levels this may take several days.

4. When the system has reached room temperature, close the dump valves M22 and M27.

5. Note date, time, cryogen levels, etc. in the log book.

3.11 Energizing the Main Magnet

This procedure describes ramping up the main superconducting magnet sitting in the LHe reservoir and holding the tail extention of the IVC and mixing chamber within its bore. It requires that there be LHe in the reservoir sufficient to cover the magnet (see Procedure 3.3), which means the level meter meter must give a positive, non-zero value.

1. Start the LabView control software for the magnet on the control computer. If not already running, start the oimenu.vi window by clicking on theOxford Instr icon on the desktop. Select the Instruments menu and click on the IPS Front Panel button.

2. Click on the persistent mode switch heater. The supply will wait 30 seconds for the switch to warm.

3. Click on the settings button and enter the desired field up to 15 Tesla and the desired ramp rate up to 0.635 Tesla per minute for fields up to 9.5T and up to 0.317 T/min for fields from 9.5T to 15T. Click OK and then click the sweep-to-field button.

The last step may be repeated as often as desired to energize the magnet at any desired field between -15 and +15 Tesla.

3.12 Entering Persistent Mode

This procedure describes putting the superconducting magnet in persistent mode and ramping down the power supply. It assumes that the magnet has been energized with Procedure 3.11 (Procedure 3.14 may have been performed as well).

1. Record the value of the current and field in the logbook.

2. Click off the switch heater. The supply will wait 30 seconds for the switch to cool and then ramp down the leads to zero current. The persistent mode light will come on.

3.13 Leaving Persistent Mode

This procedure describes ramping up the power supply and taking the superconducting magnet out of persistent mode. It assumes that the magnet has been energized with Procedure 3.11 (Procedure 3.14 may have been performed as well) and then Procedure 3.12 used.

1. Click the magnet status button to determine the field. Click on the settings button and enter this field. Click OK and then click the sweep-to-field button.

2. Click the switch heater on. The supply will wait 30 seconds for it to warm and open. The persistent mode light will go out.

3.14 Running the Lambda Fridge and Reaching Fields over 15 Tesla

This procedure details starting the lambda fridge pumping, cooling the superconducting magnet to 2.2 K, and ramping up the magnet from its 4.2 K maximum. It assumes that Procedure 3.11 has been performed to bring the magnet to 15 Tesla and that Procedure 3.12 has been used to place the magnet in persistent mode.

1. Fill the LHe reservoir with Procedure 3.6.

2. Start the LabView control software for the lambda fridge on the control computer. If not already running, start the oimenu.vi window by clicking on the Oxford Instr icon on the desktop. Select the Instruments menu and click on the Fridge Front Panel.

3. Click on the Lambda Controller.vi Start button. In response to the dialog box, start the Lamda pump and open M58. Click OKay.

After the temperature has reached 2.2 K, the Fridge Ready light will come on and the magnet may be raised to any field between 15 Tesla and its 2.2 K maximum of 17 Tesla.

1. Take the magnet out of persistent mode (Procedure 3.13).
2. Click on the settings button. Set the desired field between 15 and 17 Tesla. Set a ramp rate up to 0.159 Tesla per minute. Click OK and then click the sweep-to-field button.

The last step may be repeated as often as desired to energize the magnet at any desired field between 15 and 17 Tesla.

3.15 Stopping the Lambda Fridge

This procedure details ramping down the magnet to its 4.2 K maximum and stopping the lambda fridge pumping. It assumes that Procedure 3.14 has been performed.

1. If the magnet is in persistent current mode, perform Procedure 3.13.
2. Click on the settings button. Set the desired value to the 4.2 K maximum of 15.0 Tesla. Set the ramp rate to 0.159 Tesla per minute or less. Click OK and then click the sweep-to-field button.
3. Execute Procedure 3.12 to close the switch and remove current from the magnet leads.
4. Click on the Lambda Controller.vi Stop button.
5. Close the pumping valve M58. Shutoff the pump. If desired, the magnet may be taken out of persistent current mode by Procedure 3.13.

3.16 De-Energizing the Main Magnet

This procedure describes ramping down the main superconducting magnet sitting in the LHe reservoir and holding the tail extention of the IVC and mixing chamber within its bore. It assumes the magnet has been energized with Procedure 3.11.

1. If the magnet is in persistent current mode, perform Procedure 3.13.
2. If the field is over 15 Tesla, Click on the settings button. Set the desired value to the 4.2 K maximum of 15.0 Tesla. Set the ramp rate to 0.159 Tesla per minute or less. Click OK and then click the sweep-to-field button.
3. If the field is over 9.5 Tesla, Click on the settings button. Set the desired value to the sweep rate breakpoint of 9.5 Tesla. Set the ramp rate to 0.317 Tesla per minute or less. Click OK and then click the sweep-to-field button.

4. If the field is 9.5 Tesla or less, Click on the settings button. Set the desired value to 0.0 Tesla. Set the ramp rate to 0.635 Tesla per minute or less. Click OK and then click the sweep-to-field button.

5. When the supply reaches zero current and stops ramping, click off the switch heater button.

3.17 Energizing the Transfer Magnet

This procedure describes ramping up the transfer superconducting magnet located on the ouside of the IVC can where it extends the fringe field of the main solenoid to the 1K pot for maintaining the polarization of targets during transfers. It requires that there be LHe in the reservoir sufficient to cover the main magnet and reach the copper cylinder on which the transfer magnet is wound (see Procedure 3.3), which means the level meter meter must give a positive, non-zero value.

1. Turn on the Hewlett Packard power supply.

2. Ramp the supply up to 10 amps.

3.18 De-Energizing the Transfer Magnet

This procedure describes ramping down the transfer superconducting magnet located on the ouside of the IVC can where it extends the fringe field of the main solenoid to the 1K pot for maintaining the polarization of targets during transfers. It assumes the magnet has been energized with Procedure 3.17.

1. Ramp the Hewlett Packard Power Supply down to zero current.

2. Turn off the supply.

3.19 Target Transfer

This procedure describes the steps required to transfer a target into or out of the dilution refrigerator. It assumes the refrigerator is idling at 4.2 K (see Procedures 3.3 and 3.9). This procedure requires that the transfer cryostat be cold and positioned on its cart ready to be lifted. (see the Transfer Cryostat manual).

1. If exchange gas is present, perform Procedure 3.21 to remove it.

2. If polarization is to be retained, take the the magnet out of persistent mode (Procedure 3.13) or start the magnet (see Procedure 3.11) and move the field to 10 Tesla (see Procedure 3.11.

3. If polarization is to be retained, turn on the transfer magnet with Procedure 3.17.

4. Perform Transfer Cryostat Procedure 3.9 to attach the two cryostats.

5. Follow the Procedure for withdrawing a target, Transfer Cryostat Procedure 3.15, or inserting a target, Transfer Cryostat Procedure 3.16.

6. Perform Transfer Cryostat Procedure 3.10 to detach the two cryostats.

7. Turn off the transfer magnet with Procedure 3.18.

3.20 Adding Exchange Gas

This procedure describes the steps required to add helium exchange gas to the inner vacuum chamber space of the dilution refrigerator. It assumes the refrigerator is is idling at 4.2 K (see Procedures 3.3 and 3.9).

1. Attach a helium gas cylinder with regulator and shutoff valve to M34. Purge the line before attaching.

2. Attach a pressure gauge to M44. The gauge should be of a type which is independent of gas type and capable of reading in the few Torr regime.

3. Start the 1K pot pump. Check that valves V7, M35A, M35B, M45 and MS47 are closed. Open valves V2A, V5A, M44 and M46 and evacuate to better than 50 microns. Close valve V5A. Open M45.

4. Confirm the sorb heater is off.

5. Crack open valve M34 to the helium gas cylinder and slowly raise the pressure in the IVC to 5 torr. Close M34, M44 and M45.

6. Open V5A and evacuate the lines. Close V2A, V5A and M46.

7. Remove the helium gas cylinder, the pressure gauge and shutoff the 1K pot pump.

3.21 Removing Exchange Gas

This procedure describes the steps required to remove helium exchange gas from the inner vacuum chamber space of the dilution refrigerator. It assumes the refrigerator is is idling at 4.2 K (see Procedures 3.3 and 3.9).

1. Attach a turbo pumping station to the inner vacuum chamber pumpout, M44.

2. Power the sorb heater to 10 mW or higher to raise the sorb temperature to 30 K or higher.

3. Evacuate the target vacuum space to better than 5×10^{-6}torr through M44 and M45.

4. Close MV45 and M44, and remove the station.

5. Turn off the sorb heater and allow it to cool below 10 K.

3.22 Making HD Ice

This procedure describes the steps required to transform 30 atmosphere liters of HD gas into 30 cc's of HD ice inside a target cell. It assumes the dilution refrigerator is idling at 4.2 K (see Procedures 3.3 and 3.9).

1. Attach the Recovery and Icemaking manifolds from the Production cryostat to the gate valve atop the dilution refrigerator, M43. The vacuum lock attaches to the ISO-LF63 opening and the flexible line from RV6 and MV8 attaches to the ISO-KF16 pumpout flange.

2. Fill the LHe reservoir with Procedure 3.6.

3. Open the 1K pot needle valve N61 to 100% to fill the pot with 4.2K liquid.

4. Perform Production Manual Procedure 3.15 with the following changes to accommodate the change in dewar:

   (a) Ignore steps to change the level in the vari-temp space, which does not exist in this dewar.

   (b) References to gate valve MV2 should be to gate valve M43.

   (c) Use Procedures 3.20 or 3.21 in this manual to add or remove exchange gas.

   (d) Should it be necessary, use Procedure 3.23 in this manual to recover HD gas from a failed target production incident.

5. If no additional targets are to be made, detach the recovery manifold from the gate valve, M43, Remove the exchange gas with Procedure 3.21. Close N61.

3.23 Recovering HD Gas

This procedure describes the steps required to recover the up to 90 atmosphere liters of HD gas that may be present in the inner vacuum chamber (IVC) from the failed production of 3 targets with 30 cc's of HD ice each. It assumes the dilution refrigerator is idling at 4.2 K (see Procedures 3.3 and 3.9), that the gas recovery manifold is attached to the cryostat and that a sufficient number of 46 liter and 23 liter tanks with sufficient free volume to hold the HD in the IVC and remain below one atmosphere are available. Recall that a target has 30 atmosphere liters of HD gas so the pressure in the tank(s) must be less than 0.348 bar or 264 torr or 19.5 inches to hold a full target.

1. If helium exchange gas is present, perform Procedure 3.21 to remove the helium exchange gas from the IVC.

2. Stop the refrigerator with Procedure 3.10.

3. Blankoff the ISO-LF63 opening on gate valve M43. Attach a pump to the pumpout valve MV7. Confirm MV9 is closed. Open MV8. Evacuate the cryopump section of the manifold and the body of the gate valve through MV7 and then close it.

4. Depending on the amount of HD in the IVC and the space available in the tanks the following steps may need to be repeated several times. Attach two tanks to the tank section of the recovery manifold.

5. Close MV8. Open MV9. Evacuate the cryopump section and the tank section of the recovery manifold through MV7 and then close it

6. Open the valve(s), MV10, to the tank(s). Note the pressure and then close MV9.

7. Open M43 and MV8 to connect the IVC and recovery manifold cryopump section.

8. Determine the pressure in the IVC. If it is greater than the HD tank(s), open MV9 to connect the IVC to the HD tank(s). When the pressures equalize, close MV9. Now we must cryopump out the rest of the HD to fill the attached tanks.

9. Close MV9 and open MV8. Slowly insert the long thin cryopump tube into a liquid helium supply dewar. Monitor the pressure to determine the pumping progress.

10. When the pressure has reached 1×10^{-3} bar absolute or the tube touches the bottom of the dewar, close MV8, open MV9 and pull the cryopump tube out of the liquid helium supply dewar.

11. If the pressure in the IVC is below 1×10^{-3} bar absolute, all the HD possible has been collected; go on to the next step. Otherwise if there is more room in the current tanks, close MV9, open MV8 and go back to step 9. If there is no more room in the current tanks, you will need to change tanks. Close the valve(s), MV10,on the tank(s). Cryopump the tank section by closing MV8, opening MV9, and inserting the cryopump tube in LHe. Return the HD to the IVC by closing MV9, opening MV8, and take the cryopump tube out of LHe. Detach the tanks and go to step 4.

12. Close the valve(s), MV10,on the tank(s) and detach the tanks. Close M43 and detach the recovery manifold from the dilution refrigerator.